Appendix K

ICET Test 2: Pre-Test, Test, and Post-Test Project Instructions

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List of Tables

The ICET test series is conducted under the guidance of project instructions (PIs) that identify the steps to follow for certain activities. These PIs are revised or re-written as needed for each test. For Test 2, new PIs were written to address pre-test operations and test operations. The PI that addresses post-test operations was revised for Test 2. These three PIs are included in this appendix to describe more completely the test apparatus and chemical solution preparations, the test startup and daily sampling, and the steps followed after test shutdown.

K1 ICET TEST 2 PRE-TEST OPERATIONS

K1.1 INTRODUCTION

K1.1.1 PURPOSE

The first intent of this instruction is to ensure that all data acquisition, testing samples, testing supplies, and related material are ready and accounted for prior to testing. The second intent of this instruction is to prepare the chemical tank for testing.

K1.1.2 SCOPE

The pre-test operations preparation will ensure that successful implementation of the testing activity is achieved.

K1.1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.a, October 6, 2004
- TSP Chemical Additive Analysis, Test #2—ICET-CALC-011
- Laboratory Safety Guidelines
- ASTM A 380–99, Standard Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems
- Material Safety Data Sheets (MSDS) for all chemicals involved

K1.2 PREREQUISITES

The data acquisition setup and inspection; calibration; and the coupon receipt, preparation, inspection, and storage tasks must be completed in full prior to performance of this activity. Fiberglass samples must be weighed and their planned locations in the tank identified. That data must be entered into the fiberglass spreadsheet

K1.2.1 TRAINING REQUIREMENTS

The following personnel training is required for this task:

1. LabVIEW and computer data acquisition training
2. Chemical handling training, specifically for ethyl alcohol, ammonium hydroxide, sodium hydroxide, and lithium hydroxide.
3. Safe lift execution training

K1.2.2 EQUIPMENT REQUIREMENTS

The following equipment is required to perform this activity: computer with installed LabVIEW software, data acquisition system, and fully assembled and calibrated ICET test apparatus.

Safety equipment must be available: goggles, gloves, lab coats, eye wash station.

K1.3 DOCUMENTATION REQUIRED

A LabVIEW operational manual is required for this task. In addition, MSDSs must be available for all chemicals used.

A lab notebook must be maintained throughout the pre-test operations procedure. Contained within the lab notebook will be the date, times, description of activities, and quantities of chemicals added, number of cleanings, and physical observations of the tank cleaning and preparation procedures.

K1.4 HAZARDS

The hazards associated with this activity include tipping of the chemical tank assembly and potential injuries associated with chemical handling.

K1.5 INSTRUCTIONS

1. Ensure that all testing materials and supplies are ready and on-site. See checklist at the end of this document. Verify that eye wash station is operational. Note: The following solutions are not used in this instruction, but are to be prepared in advance of entering ICET-PI-011, "Test Operations, Test #2 (TSP at pH = 7)." After preparation, clearly label the containers with the solutions and place in an area restricted for ICET Project test use.
2. Prepare TSP Solution Batch 1.
    a. Heat about 1.5 gallons of demineralized water, add 300 g of boric acid (H_3BO_3), and stir until the boric acid is dissolved. Pour the solution into a 5-gallon plastic container. Dissolve the boric acid in multiple batches if necessary.
    b. Add additional demineralized water to the 5-gallon plastic container until it contains about 4 gallons.
    c. Dissolve 1893 g of TSP (Na_3PO_4-$12H_2O$) into the water in the container.
    d. Dilute with additional demineralized water until the volume is 5 gallons.
3. Prepare TSP Solution Batch 2.
    a. Heat about 1.5 gallons of demineralized water, add 300 g of boric acid (H_3BO_3), and stir until the boric acid is dissolved. Pour the solution into a 5-gallon plastic container. Dissolve the boric acid in multiple batches if necessary.
    b. Add additional demineralized water to the 5-gallon plastic container until it contains about 4 gallons.
    c. Dissolve 1893 g of TSP (Na_3PO_4-$12H_2O$) into the water in the container.
    d. Add 214 mL of 12.115 N hydrochloric acid (HCl) to the water in the container.
    e. Dilute with additional demineralized water until the volume is 5 gallons.
4. Prepare laboratory control sample (LCS). See the Chemical Sampling and Analysis Plan for details on the laboratory control sample.
5. Start the data acquisition system. During step 6 verify that the data acquisition system is monitoring flow rate, pump speed, temperature, and pH.

6. Flush tank.
    a. Add 100 gallons of tap water to the tank.
    b. Start pump at 25 gpm and circulate water for 5 minutes, directing water through both recirculation lines.
    c. Check for leaks. If any leaks are present, repair at this time.
    d. Drain water (see step 7). Water should be visually clear, if not, repeat flush tank sequence until water is visually clear.
7. Clean tank. Note: The tank internal will be inspected at the best method for cleaning from ASTM A 380–99 will be chosen. This may require modification to the following steps a–f.
    a. Fill tank with 22.5 gallons of tap water and 2.5 gallons of ammonium hydroxide.
    b. Start pump at 25 gpm and circulate water for 5 minutes, directing water/ ammonium hydroxide mixture through both recirculation lines.
    c. Drain tank.
    d. Fill tank with 22.5 gallons of tap water and 2.5 gallons of ethanol.
    e. Start pump as 25 gpm and circulate water for 5 minutes, directing water/ethanol mixture through both recirculation lines.
    f. Drain tank.
8. Rinse tank.
    a. Measure and record turbidity and conductivity of tap water (see Chemical Sampling and Analysis instruction sheet).
    b. Add 50 gallons of tap water to the tank.
    c. Start pump at 25 gpm and circulate water for 5 minutes, directing water through both recirculation lines.
    d. Drain rinse water. Measure and record turbidity and conductivity of rinse water. Turbidity and conductivity should be within 10 percent of initial values. If not, repeat rinse tank sequence until turbidity and conductivity are within limits.
9. Final rinse.
    a. Fill tank with 50 gallons of RO water.
    b. Start pump at 25 gpm and circulate water for 30 minutes, directing water through both recirculation lines.
    c. Drain tank.
    d. Repeat steps 9.a, 9.b, and 9.c.
    e. Drain the entire system. Repeat steps 9.a, 9.b, and 9.c. until water conductivity is less than 50 µS/cm.
    f. Drain tank.
10. Tank is now ready for testing. Proceed immediately to Instruction No. ICET-PI-004.

K1.6 ATTACHMENTS

No forms are attached to this document.

K1.7 MATERIALS CHECKLIST

_____ lithium hydroxide, 1.197 g

_____ TSP, 3.785 kg

_____ tap water supply

_____ demineralized water production system

_____ chemical handling safety equipment (lab coat, goggles, rubber gloves)

_____ analytical balance

_____ top loading balance

_____ chemical spatula

_____ six 1-gallon HDPE or PP bottles

_____ 500-mL volumetric flask

_____ 500-mL HDPE or PP bottle

_____ 2.5 gallons ethanol

_____ 2.5 gallons ammonium hydroxide

_____ turbidimeter and associated equipment

_____ conductivity meter and associated equipment

K2 ICET TEST 2 TEST OPERATIONS

K2.1 INTRODUCTION

K2.1.1 PURPOSE

The intent of the instruction is to outline the steps that are to be followed during testing.

K2.1.2 SCOPE

This activity forms the core of the entire Chemical Effects Testing project. All activities involved in this project affect and are affected by this activity.

K2.1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.a, October 6, 2004
- ASTM Standard G 4-01
- ASTM Standard D 3370-95a
- ASTM Standard G 31-72
- Material Safety Data Sheets (MSDS) for all chemicals involved
- LabVIEW operation manual
- Laboratory Safety Guidelines
- TSP Chemical Additive Analysis; Test #2—ICET-CALC-011

K2.2 PREREQUISITES

All sample coupons must be placed in their corresponding racks. Also, the pre-operation test preparation activity must be completed in full.

K2.2.1 TRAINING REQUIREMENTS

The following personnel training is required for this task:

1. LabVIEW and computer data acquisition training.
2. Chemical handling training for all chemicals involved.

K2.2.2 EQUIPMENT REQUIREMENTS

The following equipment is required to perform this activity: computer with installed LabVIEW software, data acquisition system, and fully assembled and calibrated ICET test apparatus.

Safety equipment must be available: goggles, gloves, lab coats, hard hats, steel-toed shoes, eye wash station, hydrogen detector and hydrogen removal system.

K2.3 DOCUMENTATION REQUIRED

A lab notebook must be maintained throughout the testing procedure. In addition, a binder will be maintained that includes pertinent test instructions and the completed daily log sheets (see Attachment A). The daily log sheet contains the date, times, physical description, and quantity of fiberglass and water samples obtained each day. In addition, the daily log sheet contains information from the data acquisition system (DAS), the water samples taken, and other test information.

The electronic data that are acquired daily are backed up daily and stored in a separate location each testing day. Refer to ICET-PI-001, Data Acquisition Setup and Inspection.

K2.4 HAZARDS

The hazards associated with this activity include tipping of the chemical tank assembly, ingestion and/or respiration of any chemicals involved, and scalding and/or burning hazards involved in daily tank venting, and possible hydrogen gas generation from corrosion reactions. Appropriate measures to control hydrogen gas must be in place before operations commence.

Lifting hazards associated with the tank lid and coupon racks are also associated with this activity.

K2.5 INSTRUCTIONS

1. Pre-Operation Preparation should be complete before proceeding with this sequence.
2. Ensure that all testing materials and supplies are ready and on-site (see checklist at end of this instruction).
3. Add 240 gallons of RO water to the tank by pumping water from the RO skid through the totalizing flow meter. Record flow to the nearest 0.5 gallon.
4. Verify valves are positioned as follows:

Table K2-1. Valve Positions for ICET Test 2

Valve	Description	Position
V-1	tank drain	closed
V-2	pump isolation	open
V-3	instrument loop supply	open
V-4	instrument loop discharge	open
V-5	instrument loop bypass	closed
V-6	in-line filter isolation	open
V-7	tank spray supply	closed
V-8	recirculation supply	open
V-9	sample line	closed
V-10	loop drain	closed

5. Start pump and adjust to flow rate of approximately 25 gpm.
6. Start computer, start LabVIEW, verify that flow rate, pump speed, temperature, and pH are being recorded properly.
7. Turn on heater and allow water in tank to heat to 60 °C ± 2 °C.

8. Add the pre-mixed LiOH solution.
9. Add 14.54 kg of boric acid (H_3BO_3), weighing in approx. 2 kg increments, recording the weight of each increment to the nearest 10 g.
10. Allow the water to circulate until the solution is visibly clear, indicating that the boric acid is completely dissolved.
11. Allow water in tank to heat to 65 °C ± 2 °C.
12. Take grab water sample for analysis for the parameters identified in steps a–h below. Also record physical appearance of the sample (clarity, presence of gelatinous material, etc.). All Day 1 and subsequent samples will be analyzed by Assaigai Analytical Laboratory. In addition, periodic test samples and laboratory control samples (LCSs) will also be analyzed by the UNM laboratory.
    a. pH
    b. temperature
    c. turbidity
    d. viscosity
    e. total suspended solids (TSS)
    f. dissolved oxygen (DO)
    g. chloride
    h. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved
13. Add concrete dust and latent debris samples (prepared earlier), wait 10 minutes, take 100 mL water sample for particulate size distribution, density, and TSS.
14. Stop pump.
15. Place coupon racks and fiberglass holders into tank.
16. Verify locations of coupon racks and fiberglass holders.
17. Lift tank lid into position on top of tank.
18. Start pump and adjust pump speed to 25 gpm.
19. Open valve V-7 (tank spray supply) to direct water to nozzles and adjust valves V-7 (tank spray supply) and V-8 (recirculation supply) until nozzle flowmeter is reading 3.5 gpm. Verify total flow is still 25 gpm and adjust variable frequency drive (VFD) if necessary.
20. Record date and time at which nozzle flow started. This is time t = 0 for the test.
21. Start chemical metering pump from TSP Solution Batch 1 at a rate of 0.0417 gpm (158 mL/min). The objective here and in step #22 is to add a total of 10 gallons of TSP solution in 4 hours.
22. After 2 hours, switch the chemical metering pump to TSP Solution Batch 2.
23. Take a measurement of hydrogen concentration. At 2-hour increments, repeat the hydrogen concentration measurement. If the concentration reaches 10% of the flammability limit, purge the tank atmosphere. This needs to be repeated until the hydrogen concentration has been determined to be below the flammability limit, and then the frequency of hydrogen concentration measurements is to be re-evaluated.

24. At t = 4 hours, stop the chemical metering pump and close valve V-7 (tank spray supply).

25. Immediately after closing valve V-7 (at t = 4 hours), take water grab sample for analysis for the parameters listed below. Record the time of sample collection.

    a. pH

    b. temperature

    c. turbidity

    d. viscosity

    e. chloride

    f. total suspended solids (TSS)

    g. dissolved oxygen (DO)

    h. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved

26. At t = 24 hours, t = days 2, 3, 4, 5, 6, 7, 9, 11, 13, 15, 18, 21, 24, 27, and 30 take water grab sample for analysis for the parameters listed below. Record the time of sample collection.

    a. pH

    b. turbidity

    c. viscosity

    d. temperature

    e. total suspended solids (TSS)

    f. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved. An exception is that B, Li, K, Pb, and chloride analyses will be performed only at t = days 15 and 30. Also, dissolved oxygen will be measured at day 30.

27. During each daily water sample collection, look inside tank (through windows) and record observations.

28. At t = 24 hours, and weekly thereafter and at the end of the test, collect 100 mL water sample for particulate size distribution and density analysis, to be performed at AALI. The particulate size ranges to be used will be as close as possible to those called out in the test plan: (in microns), 1-10, 11-25, 26-50, 51-75, 76-100, and > 100 microns.

29. At 14 days $\leq$ t $\leq$ 16 days and at the end of the test, collect a sacrificial fiberglass sample to be inspected and examined with SEM.

30. At 24 hours, at 14 days $\leq$ t $\leq$ 16 days and at the end of the test, run 1L of water through a nucleopore filter. The filter will be taken for SEM analysis as specified in ICET-PI-007.

31. Shut down pump

32. Indicate end of test on the data acquisition system and shut down the data acquisition software

33. Proceed directly to Instruction sheet for Post-Test Operations.

K2.6 ATTACHMENTS

K2.6.1 ATTACHMENT A. DAILY LOG SHEET

Table K2-2. Daily Log Sheet

Integrated Chemical Effects Test (Test #2)

Date: _____ Time of sample collection: _____

Sample taken by: _____ Verified by: _____

Sample bottle identification:

Assaigai (total): _____

Assaigai (filtered): _____

UNM (total): _____

UNM (filtered): _____

Control system readings:

Temperature: _____ Flow: _____ pH: _____

Analyses:

Volume filtered for TSS: _____ pH: _____

Temperature: _____ Dissolved oxygen: _____

Turbidity: _____ Viscosity: _____

Water Level: _____ Water Added: _____

Other: _____ Other: _____

Fiberglass or other samples taken: _____

Comments:

☐ Continued on back

K2.7 MATERIAL CHECKLIST

_____ boric acid, 15.14 kg

_____ lithium hydroxide (0.1 N solution), 118 mL

_____ hydrochloric acid (6 N solution), 432 mL

_____ TSP, 3786 g evenly mixed in two 5-gallon containers

_____ chemical handling safety equipment (lab coat, goggles, rubber gloves)

_____ top-loading balance

_____ weigh pan for 2 kg aliquots of boric acid

_____ 250 mL graduated cylinder

_____ 500 mL graduated cylinder

_____ totalizing flow meter

_____ sample containers (see Chemical Sampling Instruction)

_____ analytical equipment (see Chemical Sampling Instruction)

_____ pre-assembled coupon racks

_____ pre-assembled fiberglass baskets

_____ coupon handling safety equipment (hard hat, leather gloves, boots)

_____ computer disks for backup of Labview data

_____ Masterflex peristaltic pump and tubing

_____ demineralized water production system

K3 ICET TEST 2 POST-TEST OPERATIONS

K3.1 INTRODUCTION

K3.1.1 PURPOSE

The intent of this instruction is to ensure that the experimental samples are removed from the test apparatus, the test apparatus is cleaned and inspected, and the test apparatus is made ready for subsequent pre-test operations.

K3.1.2 SCOPE

This activity marks the end of one chemical effects test run. Experimental sample removals and inspections, cleaning, and preparations for subsequent tests are addressed here.

K3.1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.b, February 9, 2005
- ASTM Standard G 4-01
- ASTM Standard G 31-72
- ICET-PI-002, Coupon Receipt, Preparation, Inspection, and Storage, November 19, 2004
- ICET-PI-011, Rev. 0, Test Operations, Test #2, February 3, 2005
- ICET-PI-005, Rev. 1, Chemical Sampling and Analysis, February 3, 2005
- Laboratory safety guidelines
- ICET Project Safety Plan

K3.2 PREREQUISITES

All test operation PI criteria must be completed prior to conducting this task.

K3.2.1 TRAINING REQUIREMENTS

- Laboratory Safety Guidelines
- ICET Project Safety Plan

K3.2.2 EQUIPMENT REQUIREMENTS

A city tap water supply outlet is required for this activity and chemical handling and lifting safety equipment. A reverse osmosis unit is required for the final flush.

K3.3 DOCUMENTATION REQUIRED

Documentation related to test parameters, chemical water analyses, coupon and fiberglass examinations, and daily test operations are outlined elsewhere. In this instruction, the steps

required to remove samples from the test apparatus and to make it ready for the next test are outlined. In addition, observations as to the test apparatus' condition are obtained and recorded here.

K3.4 HAZARDS

The hazards associated with this activity include ingestion/respiration and/or dermal and eye contact with residual chemicals. Lifting hazards associated with the tank lid and coupon racks are also associated with this activity.

K3.5 INSTRUCTIONS

1. On the last day of testing, collect water samples and perform analyses as outlined in ICET-PI-011 and ICET-PI-005.
2. Remove 10L of water from the test apparatus and store at test temperature, for future analyses
3. Shut off the recirculation pump.
4. Remove the small fiberglass samples for SEM examination.
5. Leave one heater on and continue to monitor tank water temperature.
6. Isolate and drain the test apparatus piping.
7. Remove the tank lid.
8. Before removing coupon racks or insulation samples, examine and take photographs and notes of the inside of the tank, the coupons and racks, and the insulation samples.
9. Remove the six non-submerged coupon racks to a staging area for drying and post-test examinations (refer to ICET-PI-002).
10. Take additional photographs of the inside of the tank.
11. Take swipes from several locations on the inside tank wall, and place the samples in plastic holders that are marked with the sample location.
12. Drain the tank slowly, down to the level that uncovers the submerged rack, but keeping the water level above the heater.
13. Remove the submerged coupon rack to the staging area.
14. Repeat steps # 10 and 11.
15. Turn off the heater.
16. Completely drain the tank, taking precautions so that the sediment on the bottom of the tank is not disturbed any more than necessary.
17. When the tank is drained, repeat step # 10. Note especially the locations and orientations of the remaining samples.
18. Remove the remaining insulation samples to the staging area to dry.
19. Ensure that all samples removed from the tank are clearly marked as to their location and orientation within the tank.
20. After all samples have been removed, repeat step # 10.

21. Inspect the interior of the tank, noting any observations.
22. Note the presence of any sediment. Carefully remove as much sediment as possible, noting any unique aspects of it, such as location. Place the sediment in plastic containers with lids, marking the location of the sediment in the tank.
23. Remove the tank drain screen and remove the insulation sample for future analysis.
24. Rinse the tank with tap water and drain the water.
25. Fill the system with 250 gallons of tap water and circulate water through the spray nozzles and recirculation headers for at least 60 minutes. Repeat with de-mineralized water.
26. Store water that was drained from the tank until it is cleared for discharge.
27. If any signs of deterioration are observed on the inside of the test apparatus tank, remove selected insulation on the tank. Inspect the stainless steel tank for any abnormalities.

K3.6 ATTACHMENTS

No forms are attached to this document.